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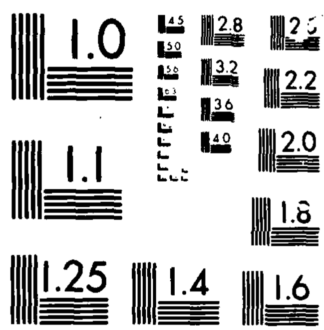

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REPORT

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A REVIEW OF METHODS TO DETERMINE THE
IGNITABILITY OF PYROTECHNIC COMPOSITIONS

L.V. de Yong

Approved for Public Release

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ABSTRACT

Ignition of pyrotechnic materials is a problem that has had relatively little attention over the years. However, many tests have been developed in an attempt to measure the ability of an energetic material to ignite from various sources. These tests range from the simple sensitivity/safety type tests (spark and impact sensitivity, ignition temperature, etc.) to the more complex techniques which measure the energy required for ignition (hot wire, laser, arc-image furnace, spark). These tests are examined in this report and comments on each of their advantages/disadvantages and their applicability to studying pyrotechnic ignition are made.

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CONTENTS

A REVIEW OF METHODS TO DETERMINE THE IGNITABILITY OF PYROTECHNIC COMPOSITIONS

1. INTRODUCTION

1.1 Background

Every piece of military ordnance contains an ignition system in which energy is transferred between two or more components in the ignition train. Such ignition trains consist of separate increments of energetic materials which may be explosives, pyrotechnics, propellants or combinations of these. With ordnance becoming more and more complex, more stringent operating requirements are demanded; not only reliable but predictable ignition and ignition transfer are of paramount importance to the proper functioning of the weapon. Problems often occur in weapons containing pyrotechnic ignition trains and solution of these is made difficult because little is known about the ignition of pyrotechnics and factors that effect ignition and ignition transfer.

Approaches to the solution of ignition problems in pyrotechnics involve a laborious study of design variables, "engineering" the problem out by modifying the design, or the use of ignitability tests. Both design variable and engineering methods attempt to identify the ignition problem in the shortest possible time and thereby improve the design to the degree necessary to meet a performance specification. Both are thus generally ad hoc approaches directed to solving the specific problem at hand. The use of ignitability type tests, however, involves basic research to study the ignition of energetic materials and define the level of stimulus required for initiation.

This paper presents a brief review of the major techniques that have been used to study ignitability of energetic materials with an emphasis on those techniques useful for pyrotechnic compositions.

1.2 Ignition and Ignitability

The process of ignition can be defined as the initiation of an exothermic reaction by increasing the temperature of a portion of the reactive material to a point where the reaction becomes self sustaining. The temperature at which this occurs is often referred to as the "ignition temperature" of the material. The ignitability of a material therefore defines its ease of ignition or the level of energy required to reach its characteristic "ignition temperature".

2. METHODS TO DETERMINE PYROTECHNIC IGNITABILITY

Most of the techniques which will be discussed below have primarily been used to measure the ability of an ignition stimulus to initiate pyrotechnics, propellants or explosives or to measure the amount of energy required to cause ignition. Although some of these techniques have not been used for pyrotechnics, they could readily be adapted for this purpose and are therefore included.

2.1 Sensitivity Testing

In order to define the sensitivity of pyrotechnic compositions, a number of hazard assessment tests are conducted. These define the ability of a composition to ignite from various stimuli and form the front line of tests examining ignition. The most frequently used tests use thermal, electrostatic or mechanical stimuli [1-4]. Only mechanical stimuli will be mentioned here as the others are examined later in the report.

Tests such as Rotter Impact, Ball and Disc and Friction Pendulum [1-4] examine the level of mechanical stimuli required to cause 50% probability of ignition. An illustration of the Rotter Impact test to determine Figure of Insensitiveness is shown in Figure 1.

Sensitivity tests are highly dedicated in that they model, or attempt to model, the working environment. As such they are principally safety tests rather than definitive ignition tests and simply provide a means of distinguishing between those materials which need a relatively high energy for ignition and those requiring a low energy. However, subject to their limitations, most of the above tests are used by researchers as screening tests for qualitative comparison purposes.

2.2 Ignition Temperature (T_i)/Time to Ignition

The ignition temperature, as defined previously, has often been used as an indicator of the ease of ignition of an energetic material and many techniques have been devised to measure it.

In the ERDE T of I method [1,4], a small sample of the test material is heated in a borosilicate test tube placed in a steel block, the temperature of which is raised at a steady rate (usually 5°C/minute) until an ignition occurs. Another variant is to determine the temperature at which the sample ignites when held at a constant temperature for a specific length of time [2]. These tests, with their arbitrary sample size and rate of heating, are essentially only stability or sensitivity tests. The results are therefore meaningful only under the particular conditions used.

More recently, ignition temperature has been determined by DSC or DTA techniques. The advantage of these techniques is that not only can the ignition temperature be determined but also the heat of reaction, activation energy, and the apparent first order Arrhenius pre-exponential term. Thus, information on both ignition and the reaction process can be obtained. However, like the previous methods, experimental conditions can substantially affect the result; the value obtained for the ignition temperature is dependent on the heating rate, and also the mass and size of the sample [2,5,6,7]. For example, Barton et al [6] showed that relatively small changes in the sample weight could radically alter the ignition temperature determined under DTA conditions. They found, for Mg/BaO_2/Acroid Resin, that changes in sample weight from 50 mg to 60 mg decreased the ignition temperature from 600°C to 350°C.

Other instrumental techniques used are thermal conductimetric analysis [8] and electrothermal analysis [9]. Both these techniques allow the temperature at the onset of ignition and the extent of the chemical reaction to be studied. They have only had limited use because of the lack of commercial instrumentation.

Johnson [5] defined the energy required for ignition of a pyrotechnic delay as:

$$E = \frac{K\rho CA^2(T_i - T_a)^2}{\pi e q_{in}}$$

K	=	Thermal conductivity
ρ	=	Density of sample
C	=	Specific heat
A	=	Area of sample
T_i	=	Ignition temperature
T_a	=	Ambient temperature
q_{in}	=	Constant heat flow in
e	=	2.718

This equation confirms the physical facts that one expects based on experience and intuition, i.e. the energy required increases with increases in specific heat, thermal conductivity, density and ignition temperature. McLain [7] rearranged Johnson's ignition equation and introduced an energy factor as a measure of the energy required for ignition.

$$(\text{Energy factor})^3 = B(T_i - T_a)^2$$

This equation allows comparison of ignitability in terms of an energy factor and the temperature of ignition. Calculated energy factors for several mixes along with the corresponding ignition temperatures are listed in Table 1. Black powder is arbitrarily assigned the value of 1.00. Experience has shown that the Pb_3O_4/Mn/Si composition is easier to ignite than black powder, which is verified by its lower energy factor. However, its ignition temperature is higher, thus the use of this parameter only as a means of defining ignition or ignitability is questionable.

The advantages of these techniques for measuring the ignition temperature are that they are fast and simple and generally have high sensitivity and reliability. The disadvantages are that the heat source is radiant and may not represent actual ignition systems. Furthermore, the results cannot usually be extrapolated to other sample sizes, densities or heating rates. Overall, the results require a large amount of interpretation.

Due to the major limitations in defining the ignition temperature, many techniques have been extended to measure the time to ignition [2,4,5,7,10-14]. In one method, a hot bath immersion apparatus is used which consists of a thin-walled metallic cup and a liquid bath of hot molten lead or Woods metal. A fixed mass of the sample (usually 1 g) is placed in the cup which is then immersed in the bath and the time lapse (induction time) between immersion and ignition is recorded. The procedure is repeated for several bath temperatures. The induction times are plotted graphically against bath temperature and the temperature for which ignition occurs within a 5 s induction time is determined [2,4,7]. A ranking of pyrotechnic ignitability can then be obtained. Strom [11] challenged the validity of values derived in this manner because in reality one is not dealing with induction times as long as 5 s. For example, a delay composition ignited with a fuse will have an induction time in the range 1-10 ms. If the temperature/induction time graphs for several compositions were extrapolated to much shorter times, it is probable that some of the times would intersect and the ranking, with regard to ignitability, might alter.

Shidlovsky [10] describes a variation of the above method wherein the induction time, as a function of bath temperature, is plotted on a straight line and extrapolated to time zero. He defines the temperature corresponding to this time as the flame point or ideal ignition temperature.

Johnson [5] suggests a method for determining the true ignition temperature using the hot bath immersion apparatus, according to the equation

$$T_i = \frac{t_1{}^2 T_{B1} - t_i{}^2 T_{Bi}}{(t_1{}^2 - t_i{}^2)} \qquad i = 1,\ 2,\ 3 \ldots \text{ number of bath temperatures}$$

T_i = true temperature of ignition
t_1 = induction time for bath temperature 1
T_{B1} = bath temperature 1

where the induction time and bath temperature of one experiment are equated with that of the next, producing an average figure for T_i. Henkin and McGill [12] extended the hot bath technique to permit the determination of activation energy from the slope of the graph of log t vs V_T.

Hot stage microscopy or hot plates have also been used to measure the time to ignition and activation energy of propellants and pyrotechnics [13,14,15]. The ignition test is performed by bringing the face of a pellet of the composition into contact with the hot plate with the time between contact and the appearance of flame being measured. The value of this data is, however, questionable. At high plate temperatures, time to ignition can be as short as a few seconds and the sample temperature may lag behind the plate temperature. Williams [16] notes that at a temperature of 600°C, after 10 seconds the sample is still about 40°C below the plate temperature. The practical relevance of the data is also questionable as the heat source is purely homogeneous i.e. solely conductive.

2.3 Capacitor Discharge : Spark Ignition

The simplest spark ignition tests are safety tests where sparks of fixed energies are used to determine electrostatic sensitivity [1,3,4]. These tests attempt to predict safe-handling conditions and define a material as either sensitive or insensitive to ignition by electrostatic discharge.

McLain and Frahm [17] attempted to generate a short lived spark of reproducible temperature by replacing the needle in a motor driven sewing machine with an electrically heated incandescent Pt-Ir filament. The power to the filament was varied until ignition occurred. Typical results are shown in Table 2 where the smaller the filament power the lower the value of ignition ease. As with previous results, there is no obvious relationship between the ignition temperatures and the ease of ignition.

Maki [18] also studied spark ignition of a range of pyrotechnics. A charged capacitor was discharged through the sample material placed in the spark gap between two electrodes (Figure 2). The energy of the spark was calculated using:

$$E = \frac{1}{2} CV^2$$

E = Spark energy
C = Capacitance
V = Voltage

The value of the ignition energy obtained is notoriously dependent on experimental variables eg. the switching mechanism, the electrode shape, the distance between electrodes, the value of the series resistance and the operator [18]. Altering the system to compare approaching and fixed electrodes produces markedly different results due to variations in the type of spark.

Although some of the problems associated with the switch have been overcome [19], these methods only produce relative ignitability figures as the system is highly dependent on many of the test components. The technique also does not reproduce actual ignition systems as no known ignition system uses spark techniques.

2.4 Arc Image Furnace

The arc image furnace uses a radiant heat energy source and has proved to be a versatile and reliable instrument in the study of ignition of energetic materials [20-24]. The method consists of concentrating the thermal radiation of an arc lamp (carbon or xenon) onto a sample with a series of ellipsoidal mirrors. A schematic of a typical arrangement is shown in Figure 3. This arrangement permits concentration of the radiant energy on a very small area and the crossing of the reflected radiation allows for easy shuttering. The heat flux intensity, energy and pulse duration can all be determined and varied independently of environmental conditions. Most of the researchers using this tool have looked at the ignition of propellants but the technique could readily be adapted to pyrotechnics.

The advantages of this technique are:

1. The flux required for ignition can be readily measured and altered and it is reproducible.

2. The energy is "clean" because it is pure radiation.

3. The sample can be irradiated very rapidly.

4. The sample can be viewed continuously throughout the test.

It does, however, have several disadvantages. These are:

1. The energy source is purely radiant and does not have any conductive or convective component. In most igniters, however, the ignition stimulus consists of conductive, convective and radiant heat transfer.

2. The arc lamp generates a broad brand spectrum which causes problems with absorption and reflection as the optical properties depend on the wavelength.

3. High heat fluxes are required to minimize heat losses.

These disadvantages make it imperative to use care when interpreting arc image results particularly for common igniter systems where conductive/convective heating is involved.

2.5 Laser Ignition

Before the early 1970s, the arc image furnace was the primary technique which used a radiant thermal energy source for studying ignition of energetic materials. A more recent method of providing radiant energy is by the use of a suitable laser. The CO_2 laser has been the most widely used [25-31,33] but Nd:Glass, Nd:YAG and Ruby lasers have also been used [32-35].

Cook and Habersat [28] utilized a CO_2 laser to conduct ignitability tests on NACO (a US Navy gun propellant). The laser operated at 10.6 μm (far IR) and delivered 400 Watts in continuous (CW) mode and 2000 Watts in pulsed mode operation, providing fluxes in the range 40 Watts/cm^2 to 400 Watts/cm^2. The energy absorbed by the irradiated sample was calculated from the laser power input and the time delay to ignition was measured. The test set up is shown in Figure 4.

Ward et al. [29] used a 50 Watt 10.6 μm CO_2 laser operating in the CW mode to provide a remote ignition source capable of igniting pyrotechnic mixes in a wind tunnel. They used the laser to study the effect of such parameters as spin rate, burn time and ignitability.

Phung et al [33] used a pulsed (0.6 ms) Nd laser to simulate events of brief energy deposition times and a CW CO_2 laser (10.6 μm) for long energy deposition times. They determined the 50% ignition probability energies for a range of thermites and intermetallic pyrotechnics. The results were compared with those obtained using a 10 kW tungsten filament arc image furnace and those calculated using a theoretical model.

Holst [31] used a 1 kW CO_2 laser (10.6 μm) to study the ignition of pyrotechnic tracer compositions for artillery rounds. He used fixed power levels (1 kW, 0.5 kW, 0.25 kW) and studied the effect of sample consolidation pressure, spin rate (up to 30,000 rpm), delay time to ignition and composition changes on the probability of ignition.

Laser ignitability techniques are being used extensively in the study of pyrotechnics both as a design tool and a diagnostic tool for in service ignition problems and evaluation of safety/vulnerability. The technique has both advantages and disadvantages. The advantages are:

1. As a radiative thermal energy source the laser has a monochromatic and coherent spectral output thus minimizing complications arising from wavelength dependent ignition behaviour.

2. High power capability (up to 2 kW).

3. Power output and flux is easily measured giving accurate correlation with GO/NO-GO results.

4. The technique is fast.

The disadvantages are:

1. At present, due to the optical properties of the irradiated sample, the technique measures only relative ignitability. These problems are particularly important when using the Nd:YAG laser (near IR at 1.06 μm). This laser has many of the properties of visible light, with reflectance being strongly dependent on surface texture, and the materials present (e.g. Mg has high reflectivity).

2. Control of the laser's spatial flux density is complex. Because of this, the irradiance intensity is not constant and the beam's flux density varies across the beam. However, there have been some attempts to overcome these problems using oscillating mirrors [25], integrating mirrors and expansion of the beam and use of the horizontal portion [28].

3. Because of the novelty of the technique, no correlation exists between the many methods used.

4. The laser ignition source does not model any "in use" ignition system as the energy source is purely radiant and does not have any conductive or convective component.

2.6 Penalty Testing

In penalty tests, a system is modified to reduce the probability that it will perform as desired. The modification is varied quantitatively to a point where a mixed response is observed (fires and no-fires) in the course of a feasible number of trials. The data can then be analysed to obtain an estimate of the relationship between the magnitude of the penalty and the probability of a satisfactory response. Extrapolation back to the design condition then yields an estimate of ignition reliability which would be obtainable in the unmodified system only with prohibitively large samples.

The earliest penalty tests were developed for explosive trains, such as the NOL Small Scale Gap Test [36] and more recently the VARICOMP technique [37]. These type of tests have only recently been applied to pyrotechnics.

Lindsley [38] used an air gap technique where the pelleted donor and acceptor pyrotechnic compositions were separated by free space (Figure 5). The donor was ignited externally and the results were reported as the distance between donor and acceptor for 50% ignition probability of the acceptor. This technique thus provides a quantitative measure of the ability of one pyrotechnic to ignite from another pyrotechnic. Williams [16] used a flash tube (0.6 mm - 2.5 mm diameter) to examine the transfer of ignition from a donor pyrotechnic composition to an acceptor (Figure 6). He studied the effect of tube diameter and acceptor pressing load on the distance for 50% ignition probability between donor and acceptor. de Yong et al. [39] also used a flash tube (13 mm diameter) to determine the "standoff distance" between a percussion primer and a range of pyrotechnic acceptors for 50% ignition probability of the acceptor. The combination of this "standoff test" with the VARICOMP theory to predict ignition reliability between percussion primers and pyrotechnic acceptors was also examined [40].

The advantages of penalty testing are the simplicity of the apparatus, the ease of computation of the mean and variance and the concentration of the testing around the mean value. The technique also models real igniter systems as the donor and acceptor can be altered to design requirements. The disadvantages are the large number of tests compared to some other techniques, the requirement to alter the penalty with each test and the comparative nature of the results. A need also exists for a standard donor and acceptor for comparative studies and for work to be done on the effect of experimental variables e.g. tube material, diameter, mass of sample etc.

2.7 Hot Wire Ignition

The ignition of pyrotechnics and propellants by hot wires is common in experimental work [41-48]. A thin metal wire is embedded in the sample such that intimate contact with the entire surface of the wire is achieved. An electric current is passed through the wire and the resulting heat is dissipated into the surrounding material by conduction. A typical experimental arrangement is shown in Figure 7. By appropriate selection of easily controlled variables (current, resistance, wire etc) the energy required for ignition, heat flux, time to ignition, and temperature of ignition may be derived.

Pantoflicek et al [41], using a pressure vessel, examined the influence of wire diameter, gas pressure, temperature and current pulse time on the ignition of propellants using copper wires (0.2 mm - 0.3 mm diameter) and currents of 50A - 500A.

Baer et al [42] used the simple apparatus in Figure 7 to study the ignition of composite propellants. They used Ni-Cr wire to examine the effects of wire diameter and heating rate on the ignition energy.

Jones et al [46] measured the critical energy and time to ignition for several pyrotechnic/primary explosive compositions using modified fuseheads. They used fixed currents to study the effect of wire diameter, wire composition (Pt,Sn,Pb,Cu) and time to ignition.

Kirkham [48] studied hot wire ignition and exploding wire ignition of a large range of pyrotechnics using a capacitor discharge system [0.25 μF charged to 0.4 - 7 kV) with various diameter platinum bridgewires.

One of the principal advantages of this technique is its experimental ease and convenience. Input energies and fluxes can be easily and finely controlled over a very wide range. The source of ignition energy is also "clean" in that heat is transferred to the sample by conduction only. This idealised situation allows easy mathematical modelling using conductive heat transfer theory. However, the simplicity and idealised nature of the testing is also a disadvantage as the conditions are very dissimilar to those present during ignition of real pyrotechnics or propellants. Also, in these tests, the ignition is initiated within the bulk of the material and not on an exposed surface as in real ignition systems. Conductive heat loss may occur from the ends of the bridgewires and, with long ignition times, the conductive heat transfer may be impaired by the evolution of gas at the wire/sample interface.

3. CONCLUSION

This report has briefly examined the main techniques that have been used to study the ignition mechanism of pyrotechnics and/or propellants. Many other techniques exist (ignition via convective heating [49,50], shock initiation [51], through bulkhead initiation [16,52]) but have not been discussed because of their more specialized nature.

Some of the methods provide data of a general nature giving valuable background information but requiring careful interpretation. Other methods employ readily controlled and measured input energies and well defined heat transfer conditions. These tests are, however, highly idealised as they rely on only one source and type of heat input. In reality, most ignition systems rely on a combination of heat sources (conductive, convective or radiative) and the trend is towards experiments that approximate more closely to real situations. This is achieved using heat flux, energy heating time etc of the same order as that observed with real igniters e.g. laser ignition tests. Real heat transfer processes may also be modelled by using appropriate test hardware e.g. penalty tests.

All of these tests help in understanding ignition and are a means of determining the ignitability of energetic materials, but we still have a long way to go.

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TABLE 1

Ignition Temperatures and Energy factors for some pyrotechnics [7]

Composition	T_i (°C)	Energy Factor
Black Powder	321	1.00
Pb_3O_4/Mn/Si	458	0.36
B/PbO_2/Viton	300	0.61
B/$BaCrO_4$	655	23.6

TABLE 2

Ignition sensitivity to hot filament [17].

Mixture	Composition %	Filament watts	Ignition Temperature °C	Ignition Ease 1 (easiest) - 6
Sulfurless meal powder	90 KNO_3 10 charcoal	2.46	400 (approx)	1
Red lead starter	90 Pb_3O_4 10 Si	2.86	555	2
Red lead starter	54.2 Pb_3O_4 34.2 Mn 11.6 Si	2.86	540	2
Litharge silicon	78.4 PbO 19.6 Si 2.0 Fuller's earth	3.25	621	3
British starter mix	54 KNO_3 40 Si 6 charcoal	3.30	560	3
Black powder, A5		3.95	457	4
British thermite mix	65 British starter mix 22 Fe_2O_3 13 Al (grained)	5.49	545	5
Red lead starter	48.1 Pb_3O_4 48.1 Mn 3.8 Si	6.33	625	6
Red lead starter	78.1 Pb_3O_4 20.8 Mn 1.1 Si	6(approx.)	-	No ignition

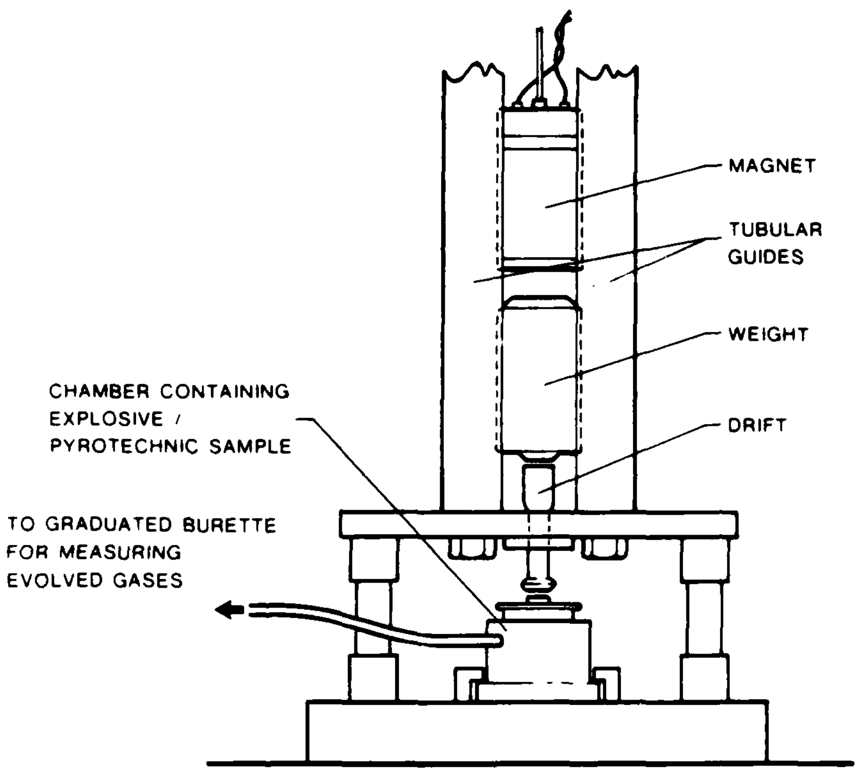

FIGURE 1 Rotter impact test for determining figure of insensitivness [1].

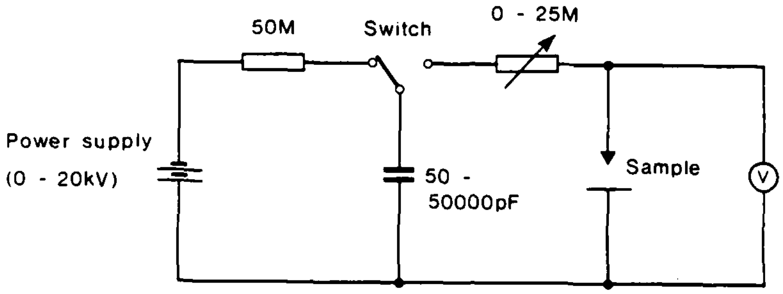

FIGURE 2 Apparatus for determining spark energy for ignition of pyrotechnics [18].

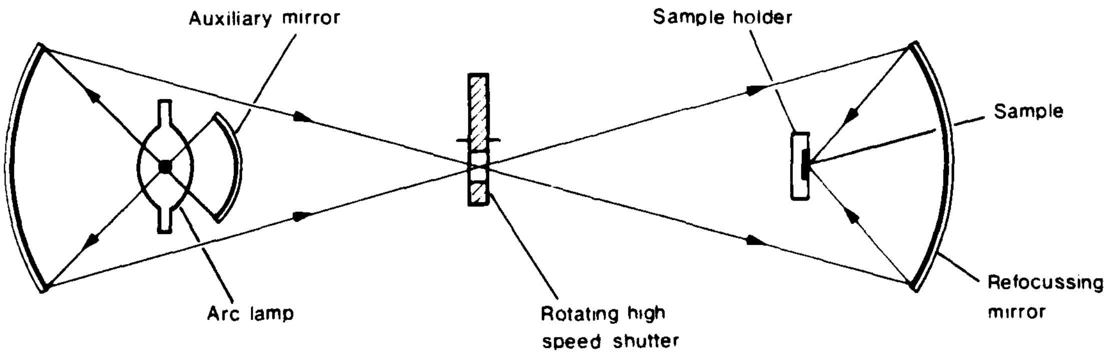

FIGURE 3 Illustration of the arc-image furnace [20].

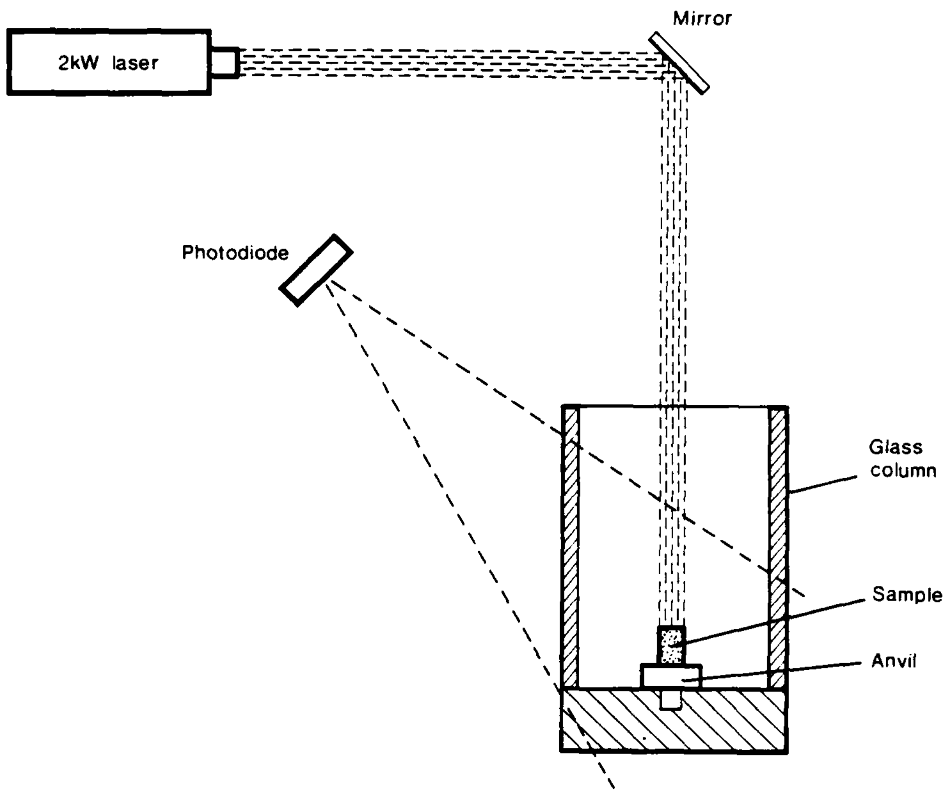

FIGURE 4 Illustration of CO_2 laser ignition of propellants [28].

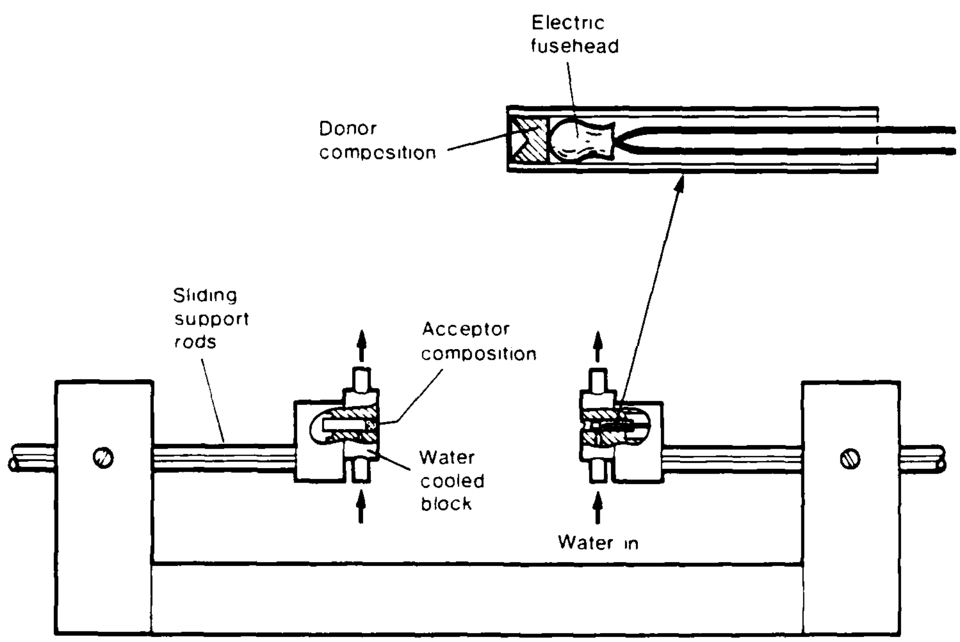

FIGURE 5 Apparatus for penalty testing of pyrotechnics [38].

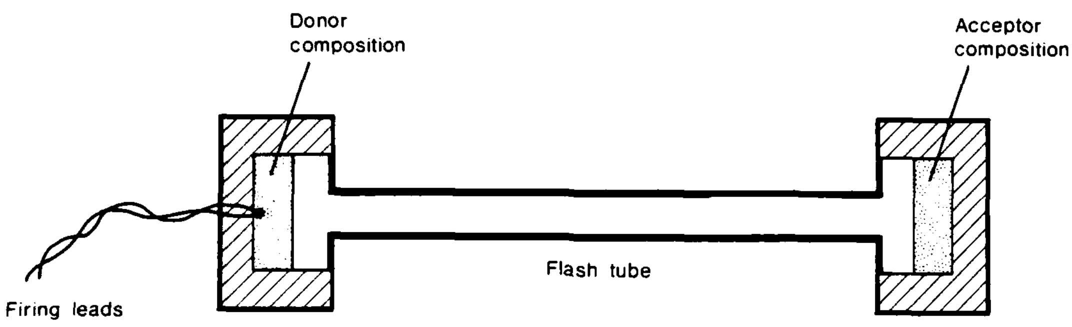

FIGURE 6 Apparatus for penalty testing of pyrotechnics using a flash tube [16].

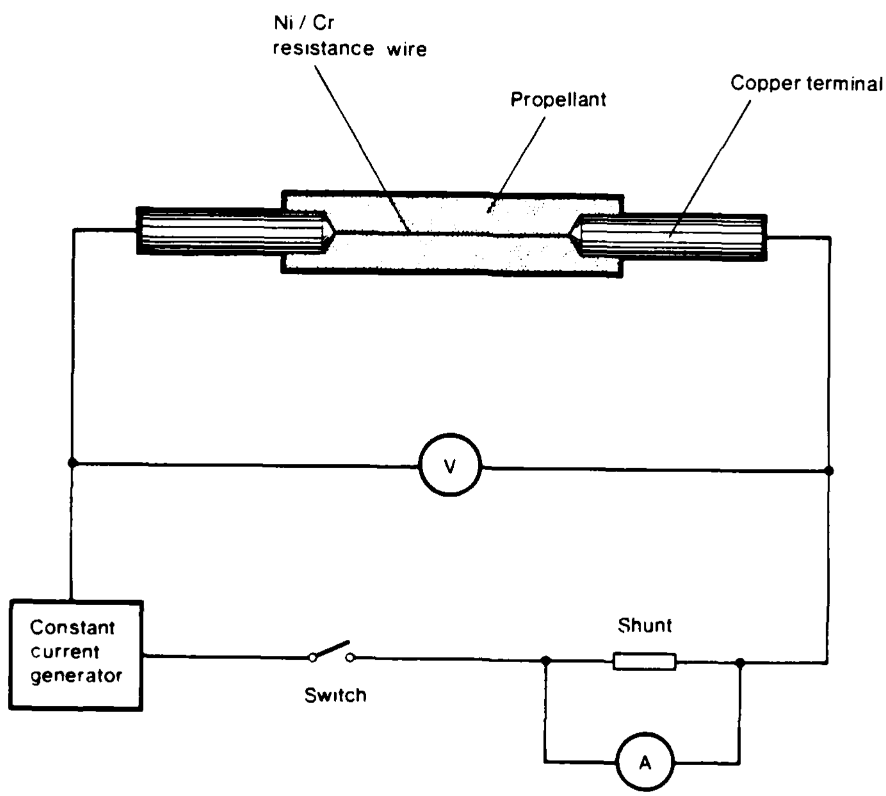

FIGURE 7 Schematic of hot wire ignition apparatus [42].

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